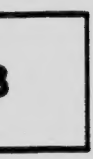

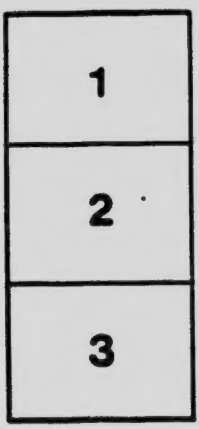

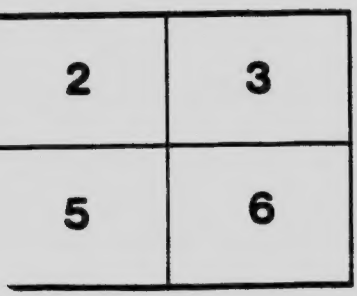

UNIVERSITY OF TORONTO
STUDIES

PAPERS FROM THE PHYSICAL
LABORATORIES

No. 68: A CONTINUOUS FLOW APPARATUS FOR THE PURIFICATION OF IMPURE HELIUM MIXTURES, BY E. EDWARDS AND R. T. ELWORTHY

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A Continuous Flow Apparatus for the Purification of Impure Helium Mixtures.[1]

By E. EDWARDS, M.A., B.Sc., and R. T. ELWORTHY, B.Sc.

Presented by PROFESSOR J. C. MCLENNAN, F. R. S.

(Read May Meeting, 1919.)

INTRODUCTION

The utilization of the absorbent property of cocoanut charcoal, cooled to a low temperature, introduced by Dewar[2] primarily for the production of high vacua and later for the separation of gases, has been practically the only laboratory method adopted for the isolation and purification of helium.

The usual practice is to admit impure gas gradually at low pressures to the charcoal contained in vessels immersed in liquid air and as soon as equilibrium is attained the unabsorbed helium is pumped off.

Ramsay[3] made use of this method for the final purification of helium obtained by fractional distillation of liquid air and in his examination of the rare gases evolved from the mineral springs at Bath. It was used by Moureau and Lepape[4] in their analyses of rare gases given off from French mineral springs, by Cady and McFarlane[5] in work on the helium content of natural gases in Kansas, U.S.A., and by Professor J. C. McLennan and others in an investigation of the helium resources of the British Empire.[6]

Two hundred litres of helium prepared from monazite with which Professor Kammerling Onnes[7] carried out determinations of isothermals—work which culminated in the liquefaction of helium—was purified by charcoal cooled in liquid hydrogen. Until recently the gas at Leiden constituted the greatest supply of pure helium existent,

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[1] Communicated by Professor J. C. McLennan, F.R.S., by permission of the Admiralty.

[2] Dewar. *Proc. Roy. Soc.*, 1904. A 74, 122.

[3] Ramsay. *Proc. Roy. Soc.*, 1905. A 76, 111.
Proc. Roy. Soc., 1908. A 80, 599.
Chem. News, 1912–105–134.

[4] Moureau and Lepape. *Comp. Rend.*, 155, p. 197–1912.

[5] Cady and McFarlane. *Jour. Amer. Chem. Soc.* 1907–29–1523.

[6] Vide this Journal.

[7] K. Onnes. *Proc. K. Akad. Wetensch Amsterdam*, 1908–11–168.

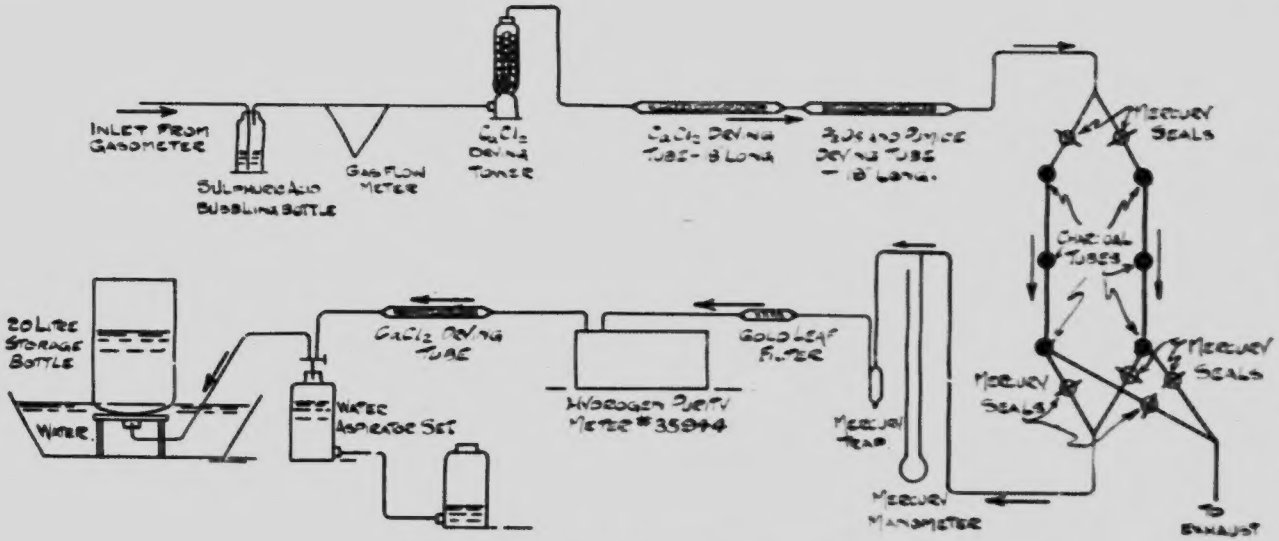

Figure 1.—Diagrammatic Sketch of Flow Apparatus.

a fact sadly referred to by the author[1] of a German aeronautical book published in 1914, after pointing out the great advantage of using helium as a balloon gas.

On account of the great advances which have now been made in the commercial separation of helium from natural gas, more rapid laboratory methods of purification are required.

Most of the work on the absorptive power of charcoal has been carried out at low pressures. Dewar,[2] however, investigated the volumes of various gases absorbed at atmospheric pressure, the heat evolved during the absorption and the rate of absorption.

Claude[3] published data on the charcoal absorption of nitrogen, hydrogen, neon and helium at pressures varying from a few hundredths of a millimeter to 247 mms. He found that the volumes absorbed increased with pressure although a considerable time was required for the complete establishment of equilibrium at the higher pressures.

In view of this work it was considered probable that cooled charcoal would be effective in removing nitrogen from impure helium, passing over its surface at atmospheric pressure at a slow rate.

As a result of successful preliminary experiments the apparatus described in the following paper was constructed.

It consisted essentially of two sets of charcoal tubes arranged in parallel so that when one set became saturated with impurity the gas flow could be diverted through the second set while the first set of tubes was revivified.

Description of Apparatus

The diagrammatic sketch, Fig. 1, indicates the general arrangement of the apparatus. The impure gas passed from the gasometer (*a*) through a Venturi meter to approximately measure the rate of flow (*b*) through a drying train into (*c*) either set of charcoal tubes as desired, (*d*) past the side tubes leading to the exhaust pump, to (*e*) a Shakespear Hydrogen Purity Meter, which, calibrated for helium[4], showed the purity of the treated gas and through it (*b*) into the receiving bottle and finally into the storage bottles. The purity was also checked by density measurements.

The appearance of the central portion of the apparatus is shown in Fig. 2, which represents the two sets of charcoal tubes arranged in parallel and the six mercury seals used instead of glass stop cocks

[1] G. Austerweil. *Die Angewandte Chemie in der Luftfahrt.*
[2] Dewar. *Loc. cit.* Also Proc. Roy. Inst. 21–240–1914.
[3] Claude. *Comptes Rendus.* 1914, 158, 861.
[4] V. F. Murray, *vide* this journal.

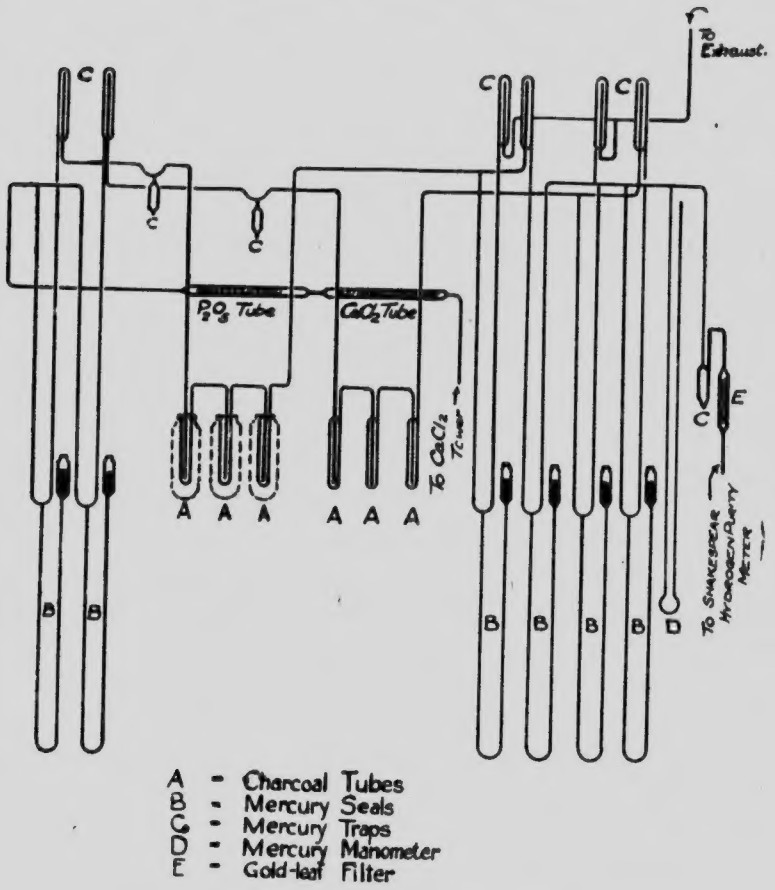

Figure 2.—Central Portion of Flow Apparatus.

to preclude the possibility of air leaks at taps. These seals were operated by lowering or raising the mercury reservoirs according as it was desired to open or close a path to the gas flow.

For use the whole apparatus was first exhausted; the charcoal tubes were surrounded by liquid air and gas was allowed to stream slowly in until atmospheric pressure was attained. When a steady condition was obtained, a flow of gas was started, and continued so long as the purity meter read above 99·8%. When this percentage began to fall the gas stream was diverted through the second set of tubes and the purification continued. Meanwhile the charcoal in

the first set of tubes was revivified for use, when the second set became saturated.

The Preparation of the Charcoal

The charcoal used in the tubes was prepared by heating fragments of cocoanut shell out of contact with air in a muffle furnace at a temperature of about 800°C. for ten hours. When cool, the pieces were crushed and screened, that which passsed 10 but was retained by 30 meshes to the inch, being used.

After being put in the tubes the charcoal was again heated in vacuo. Each tube held about 30 grams.

The Revivification of the Charcoal

When saturated the tubes were allowed to warm up to atmospheric temperature while the gas given off was withdrawn. The tubes were raised to about 200°C. by means of cylindrical electric heaters and after half-an-hour a vacuum pump was connected and the tubes thoroughly exhausted and allowed to cool. The tests have shown that the absorptive power of the charcoal is not decreased by successive treatments, but is rather increased, a fact confirmed by Lemon[1], working on the critical temperatures at which absorbed gases are evolved from charcoal.

Results

Altogether, seven runs were made, with the object of testing the efficiency of the method, the quantity of gas that could be purified by the amount of charcoal used (90 grams), the maximum rate the gas could be passed through the apparatus, and the efficacy of the revivification process. The following particulars outline the salient features of the runs:

No. of Run	Set of Charcoal tubes used	Time occupied Hours	Amount of pure gas obtained. Litres	Rate of flow. Impure gas in litres per hour	Purity %
I	1	5·62	11·1	2·38	99·9
There was no indication that the charcoal was saturated when the run was stopped.					
II	1	5·27	17·6[1]	4·15	99·9
	2	0·83	3·9	5·60	99·9
At the end of Run I the charcoal tubes of Set 1 were only pumped out by means of aspirator bottles, accounting for low figure for saturation.					
III	1	3·27	23·6[1]	8·65	99·9
	2	1·5	7·0	5·60	99·9
IV	1	3·82	21·3[1]	6·95	99·9
	2	1·05	7·2	8·20	99·9
V	1	2·00	16·7	10·0	99·9
VI	1	3.16	25·0[1]	9·50	99·9
VII	1	2·15	21·2[1]	10·9	99·9

[1] Signifies that the charcoal was saturated.

[1] Lemon. *Physical Review* 9, p. 336, 1917.

The first set of charcoal tubes was given the most complete trial. The following volumes of pure gas were recovered before the charcoal was saturated.

Run III	23·6 litres
" IV	21·3 "
" VI	25·0 "
" VII	21·2 "
Mean	22·8 "

The impure gas contained 88% "C" gas, therefore 26 litres of impure gas was treated on an average. Ninety grams of cocoanut charcoal was saturated by 3 litres of impurity (in this case nitrogen). The partial pressure of the nitrogen was about 90 mms. From Claude's data 11·2 litres of nitrogen was absorbed at 90 mms. by 100 gms. of charcoal, but the gas was left in contact with the charcoal for hours. In these experiments the impure helium was in contact with the charcoal for from one to two minutes only. It is probable that charcoal in a more efficient condition could be prepared, resulting in an even greater volume of gas being passed through before the charcoal becomes saturated.

Summary

The results of the tests show:

1. That helium containing at least 12% impurity can be readily purified by passing in a continuous stream over charcoal at the temperature of liquid air or oxygen.

2. The rate of flow can be increased up to at least ten litres an hour without decreasing the efficiency of the process. The maximum possible rate of flow was not determined.

3. By the use of two or more sets of charcoal tubes, arranged in parallel, the process may be made a continuous one as this form of apparatus permits of revivification of the charcoal without disturbing the flow.

This work was carried out at the Admiralty Physical Laboratory, South Kensington, under the direction of Professor J. C. McLennan, F.R.S.

UNIVERSITY OF TORONTO STUDIES

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